THE REMOVAL OF ILMENITE-WATER BASED FILTER CAKE IN HPHT WELLS

BY

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Dedication

To my lovely mother, my family, and my friends for their endless love and spiritual support in my life.

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Above all, I give thanks to Allah for his uncountable blessings.

This work would not have been possible without contributions from several individuals and organizations. Their help, effort, and support had been substantially valuable throughout my graduate studies at king Fahad University of petroleum and minerals.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

HPHT: High Pressure High Temperature

DTPA: Di-ethylene Tri-amine Penta acetic Acid

EDTA: Ethylenediaminetetraacetic acid

GLDA: L-Glutamic acid N, N-diacetic acid

XRD: X-Ray Diffraction

SEM: Scanning Electron Microscope

Wt.%: Weight Percentage

ABSTRACT

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The primary goal of drilling a well is to enhance the productivity or injectivity by placing the well in the optimum reservoir zone. This goal can be achieved by using a proper drilling fluid. Drilling fluid design is very important aspect of drilling operations to avoid any future challenges of the well future life such as filter cake forming, and formation damages. Thins filter cake forming is the main drilling fluid properties, and filter cake removal techniques plays a key role in drilling and completion operations. Drilling in critical downhole conditions such as high pressure-high temperature environment requires additional special type of drilling fluid because of the complexity of these conditions. Ilmenite has been evaluated as a good drilling weighting material in such conditions

The main objective of this study is to evaluate the removal problems of ilmenite water-based drilling fluid to overcome difficulties related to HPHT drilling fluids. Several experiments have been performed in the lab to determine the solubility of several chelating and chemical agents on the ilmenite particles at HPHT under dynamic condition. Water-

based drilling fluids with a density of 98.6 pcf. containing ilmenite (5 µm) were used to conduct the filtration test.

The water-based drilling fluid rheological properties, the filtrate volume, and the thickness of the filter cake were determined at HPHT condition.

HPHT filtration tests were performed at 250°F and 300 psi on Indiana limestone cores with an average porosity of 12% and an average permeability of 20 mD under static conditions. New fluid formula was developed to remove ilmenite filter cake. Afterwards, filter cake removal experiment was performed to study the impact of using a recently developed formulation on the limestone core sample and calculate the retained permeability.

All the used chelating agents such Di-ethylene Tri-amine Penta acetic Acid (DTPA), Ethylenediaminetetraacetic acid (EDTA), and L-Glutamic acid N, N-diacetic acid (GLDA) have reduced the solubility of ilmenite particles. GLDA at low pH (4), EDTA at low pH (7), and DTPA at pH (5) showed 27%, 11%, 20% solubility of FeTiO3 particles at 300°F respectively. However, when using GLDA 15 wt.% with DTPA 20 wt.%, the ilmenite dissolution rate has increased to 35%. In addition to that, 1 wt.% of Hcl has increase the ilmenite dissolution rate to 43% when used with 15 wt.% of GLDA.

The new effective solution is Green Biodegradable Removal Formulation (GBRF) where is was able to dissolve 69% of the ilmenite at HPHT condition after 24 hours' period. This GBRF solution (75 wt.% in a 300 g solution) was able to remove the filter cake introduced by water-based drilling fluids weighted with ilmenite particles. The new system has 100%

removal efficiency of filter cake using Indiana limestone core. The average returned permeability for the tested cores was about 100% for the same core.

ملخص الرسالة

الاسم الكامل: ناصر مهدي عبيان الخنفري القحطاني

عنوان الرسالة: إزالة ترشيح مادة الإلمينيت بكفائة عالية في الأبار ذات الضغط و درجات الحرارة المرتفعة

التخصص: هندسة البترول

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الهدف الرئيسي من حفر البئر هو تحسين االأنتاج وذلك بوضع البئر في المكان الأفضل على الإطلاق في المخازن الجيدة. للحصول على هذي النتائج يجب استخدام سوائل الحفر الجيدة و اللتي تساعد على الوصول الى الهدف المطلوب و ذلك يعتمد على تصميم سائل الحفر و هو جانب مهم جدا من عمليات الحفر لتجنب أي تحديات مستقبلية من حياة البئر الإنتاجية.

تعتبر عملية تشكيل الترشيح على جدار البئر أثناء عمليات الحفر هي من خصائص سوائل الحفر الرئيسية ، وتلعب تقنيات إزالة الترشيح دوراً رئيسياً في عمليات الحفر والصيانة وكذلك اختيار المادة المصعنة لسائل الحفر . يتطلب الحفر في ظل الظروف الحرجة مثل الضغط المرتفع ودرجة الحرارة العالية نوعا خاصا من سوائل الحفر نظرا لصعوبة هذه الظروف و تعتبر مادة الإلمينيت ملائمة لهذه الظروف .

الهدف من هذه الدراسة هو تقييم مادة الإلمينيت كمادة مستخدمة في صنع سوائل الحفر في درجات الحرارة العالية والضغط المرتفع. العديد من التجارب اجريت في هذي الدراسة لمعرفة قابلية ذوبان مادة الإلمينيت في بعض المواد المخلبية و الكيميائية المختلفة في نفس الظروف . و تم استخدام سوائل حفر ذات قاعدة مائية وتحتوي على الإلمينيت لإجراء اختبارات ترشيح .

كذلك تم استخدام مادة (الجي بي ار اف) للمرة الأولى في تاريخ صناعة الطاقة لإزالة الترشيح الناتج من استخدام سائل الحفر المضاف اليه مادة الإليمينيت.

المادة الجديدة المستخدمة في ازالة الترشيح اثبتت فاعليتها بشكل كبير جدا و لم تسهم فقط في ازالة الترشيح بل قامت كذلك بزيادة نفاذية الصخور المستخدمة في الأختبارات, و كذلك لم تظهر اي اثار جانبية عكسية عند استخدام المادة الجديدة .

CHAPTER 1
INTRODUCTION

1.1. Background and Motivation

Drilling wells in the petroleum industry comes throw several processes. The most critical one is the drilling across the pay zone sections of the reservoir. Drilling these sections introduces a filtration flow into the reservoir which builds a cake over the wellbore face of the rocks.

Drilling fluids used in drilling mainly impact the well drilling success and its associated costs. The drilling fluid density affects both the rate of penetration and borehole stability. In addition to that, the drilling fluid viscosity and flow rate affect the cutting transport and wellbore cleaning. Fluid properties deeply impact the rig time needed to drill the total depth (Bourgoyne 1991; Darley and Gray 1988).

The drilling engineer is concerned with the selection and maintenance of the drilling fluid, and drilling-fluid properties because of its relation to most drilling operational problems.

Drilling fluids are designed to minimize the damage of the producing intervals by:

- Forming thin, impermeable layers named filter cakes.
- Supporting the wellbore during completion phase.
- Providing efficient cleanup after drilling and completion.

The most important factors that should be considered for drilling fluid design are following:

- Proper filter cake formation.

- Maximizing the reservoir return permeability.

The addition of each weighting material to drilling fluid created the need for reliable drilling fluid functions. Haaland et al. (1976), weighting materials are components that are usually added to the drilling fluids dissolved or suspended to provide a homogeneous fluid column (increase density) that can help fluids to perform all kinds of drilling fluid functions. The two primary sources of solids in drilling fluid are chemical additives (weighting materials) and formation cuttings Burnett & Hodge (1996). Some of these chemicals are barite (BaSO4), Calcium carbonate, manganese tetroxide (Mn3O4), Hematite (Fe2O3), and ilmenite (FeTiO3).

Ilmenite was initially used as a weighting material in the drilling fluids on 1979. Ilmenite was used as an alternative weighting material to avoid shortage, high cost removal, of barite Blomberg (1984) and Rae et al. (2001). Ilmenite (5 μm), is more stable than barite in high temperature and can overcome the equivalent circulating density (ECD) challenges in some drilling situations such as deviated wells. Haaland et al. (1976) in his extensive study illustrate some ilmenite properties such as solubility in acid, high specific gravity, reaction with sour gases and availability of large quantity. Ismail et al (1999) showed that ilmenite could produce the same mud weight as barite with smaller quantity, which yields lower solids content. The ilmenite has lower fluid loss than barite which lead to formation damage reduction. Elkatatny et al. (2013) introduced using the hydrochloric acid to remove the ilmenite filter cake in water-based. Hcl (10 wt.%) showed good dissolution about 75 wt.% of the iron form ilmenite particles after 10 hours at a temperature of 300°F. It is also illustrated that the removal of ilmenite water-based cake procedure is simpler comparing with barite water-based filter cake

In this study, an extensive work of evaluating the several chelating agents, chemicals solubility effect on the ilmenite particles in high temperature and pressure condition. This study introduces a new fluid formula to remove ilmenite filter cake efficiently. It also provides a comprehensive study of the removal efficiency of the new fluid system on ilmenite filter cake and calculating the retained permeability on Indiana limestone core sample.

1.2. High Pressure High Temperature (HPHT) Wells

The wells can be classified as high pressure-high temperature if a hole pressure gradient more than 0.8 psi/ft. and the temperature higher than 300 ^{0}F, (Schlumberger, n.d.; Smithson, 2016). In HPHT drilling applications especially drilling deep wells, it is essential to have drilling fluid that has high density and excellent thermal stability to overcome the reservoir situations, Wang et al. (2012). It also requires a special filter cake removal solution as well.

1.3. Drilling Fluid Properties, Functions, Categories, and Chemistry

The usage of drilling fluids impacts all the critical drilling parameters. Drilling fluid has been used for cooling, cleaning lubricating the bit, and continuously affecting the fragments transport.

1.3.1. Drilling fluid properties and functions

- Mud Density:

  The pressure of mud column is a function of drilling fluid density and height. The drilling fluid density maintain the pressure in borehole column to equalize both formation pressure and mud column pressure. It also influences the rate of

penetration and the ability to develop a layer on the wellbore (filter cake) that affect the wellbore stability and support. However, having extra mud weight is neither practical nor economical. It causes low rates of penetration and fracturing weak formations. Different downhole conditions such as the depth, type of formation, and local structure etc. must be considered in the choice of each mud.

Oil Field Familiarization Training Guide, Baker Hughes. (May1996)

- Viscosity

  The ability of drilling fluid to carry cuttings up the borehole and be able to suspend them when circulation is stopped. This property is measured using a Marsh Funnel and the measurement unit is sec/qt. (seconds per quart). This value can range from 20 to 80 but is normally maintained between 40 and 50. Oil Field Familiarization Training Guide, Baker Hughes. (May1996).

- Gel Strength

  Gel strength is the ability of the drilling mud to change into a gel once circulation stopped. It maintains the cuttings suspension and mud solids, while they are down the hole and not allowed cuttings to settle around the drilling bit when circulation is paused.

  In general, gel strength should be minimized in order to: -

  - Remove cuttings to the surface easily.
  - Remove formation gas to the surface.

  The gel strength is determined with a Fann VG (Viscosity/Gel) Meter and its measurement unit is lbf/100ft2 (pounds per 100 square feet). The normal drilling

fluid gel strength is between 5 and 30 lbf/100ft2. Oil Field Familiarization Training Guide, Baker Hughes. (May1996).

- Additive content

  The additive materials will lead drilling fluid to be able to cool and lubricate the bit and drill string. However, normally requires additional mud characteristics that can be introduced by adding chemicals, oil and other materials. (Oil Field Familiarization Training Guide May1996).

- Fluid loss

  Fluid loss is the drilling fluid ability to create a thing tough impermeable filter cake against the permeable portions of the borehole. Oil Field Familiarization Training Guide, Baker Hughes. (May1996).

1.3.2. Drilling Fluid Categories

Bleier (1990), declare that variety of principle play the major role of drilling fluid selection which are the following: -

- The target formation characteristics and properties such pressure, temperature, and loss zones etc.
- The water source and quality that to be used in drilling fluid and the different chemical additives.
- The environmental considerations.

Drilling fluids are categorized based on their continuous phase as following; Water-based fluids, oil-based fluid, and Pneumatic (gas) fluids.

Water-based drilling fluids are the most common used fluids. Water-based muds (WBMs) is globally used for their costly effective and environmentally friendly. The WBMs density can be improved by adding calcium carbonate. Mitchell & Miska (2011). More importantly; Melbouci (2006) introduced a new chemical additive that overcome the main WBM drawback while drilling at high temperature areas, which is the sever variation in rheological and filtration properties that caused by the thermal degradation of WBMs chemical additives.

Oil-based muds are more expensive and require more environmental considerations. OBMs usually used in rough drilling environments, such as deep, horizontal and extended wells due to its ability to reduce the well friction. However, it has expensive disposal method and has low HSE profile. Mitchell & Miska (2011).

The use of pneumatic drilling fluids is limited to depleted zones or areas where the formations are low pressured. Foaming agents are added to remove minor inflows of water Darley and Gray (1988). Fluid. Pneumatic drilling fluids are non-damaging to productive formations.

1.3.3. Drilling Fluid Chemistry

The list of chemical additives used to develop functions of the drilling fluids categorized in three basic drilling fluid types; water/clay muds, Oil/water clay muds, or Compressed gases

- Water/Clay Muds

  Currently, the most used drilling fluid type is the water/clay fluid that mainly contains of a base liquid phase of water. The clay materials could be reactive or

non-reactive solids that added to the drilling fluid to introduce its special properties. The three main components of every water-based mud system are water and reactive and inert solids. Oil Field Familiarization Training Guide, Baker Hughes. (May1996).

1. Water

   Fresh water or salt water may be used as a base phase in water-based muds. Seawater is mostly used in offshore drilling operations. Salt might be added to water to prevent dissolving and causing washouts in sensitive formations such as for drilling thick evaporite sequences. (Oil Field Familiarization Training Guide May 1996).

2. Reactive Solids

   a) Clays

      Clays are the major component of water/clays drilling fluids that affect the viscosity, gel strength and water loss of the drilling fluid and are commonly referred as gel. For example, Bentonite, Attapulgite.

   b) Dispersants

      Dispersants addidives introduce a reduction in the attraction between particles and therefore reduce the viscosity by adsorption onto clay particles. Quebracho, phosphates, lignite, tannins, and lignosulphonates are the examples of dispersants.

   c) Filtration Control Additives

      These agents develop a firm impermeable filter cake on the formation and regulate the water loss into permeable formations, due to the pressure

differential. Example of filtration loss agents are starch, sodium carboxy-methyl cellulose, polymers.

d) Detergents, Emulsifiers and Lubricants

These chemicals are used for lubrication and assist the cooling functions of the drilling fluid.

e) Defoamers

These materials avoid fluid foaming at the surface in treatment equipment.

f) Sodium Compounds

Sodium decreases the yield of the clays.

g) Calcium Compounds:

Prevent formation clays from hydrating or swelling.

3. Inert Solids

a) Lost Circulation Material (LCM) These materials are used to prevent losing points and bridge the formation. These materials have different particle sizes and come in different types. Some of these materials are fibrous, granular, flakes etc.

b) Antifriction Material:

These materials are added to the drilling fluid system to minimize torque and decrease the possibility of differential sticking. It is normally used on high angle directional wells. The most frequently used material is inert polyurethane spheres.

c) Weight Material

Weighting materials are used to control the drilling fluid density. They are high-density finely ground materials. Common weight materials are barite, hematite, calcium carbonate and manganese tetroxide. (Oil Field Familiarization Training Guide, Baker Hughes. (May1996).

1.4. Filter Cake formation, Properties and Removal Methods

1.4.1. Filter cake Formation

In many drilling operations across in situ hydrocarbon reservoirs, cake filtration happens in these reservoirs. Fracturing of the rock and overbalanced drilling of wells into petroleum reservoirs are examples of the processes that introduce a cross-flow filtration, which creates a filter cake buildup.

The filtration process normally occurs under static or dynamic conditions. Static filtration exist when there is a differential pressure between wellbore and formation pressure and a continuous increase in cake thickness; or dynamic filtration, which the cake thickness is introduced from an equilibrium between solid particle deposition and erosion rate respectively Calcada et al. (2010)

A representation of the formation of a filter cake over a surface, is shown in Figure 1-1 and Figure 1-2 shows the basic, linear cake filtration process Civan (2007).

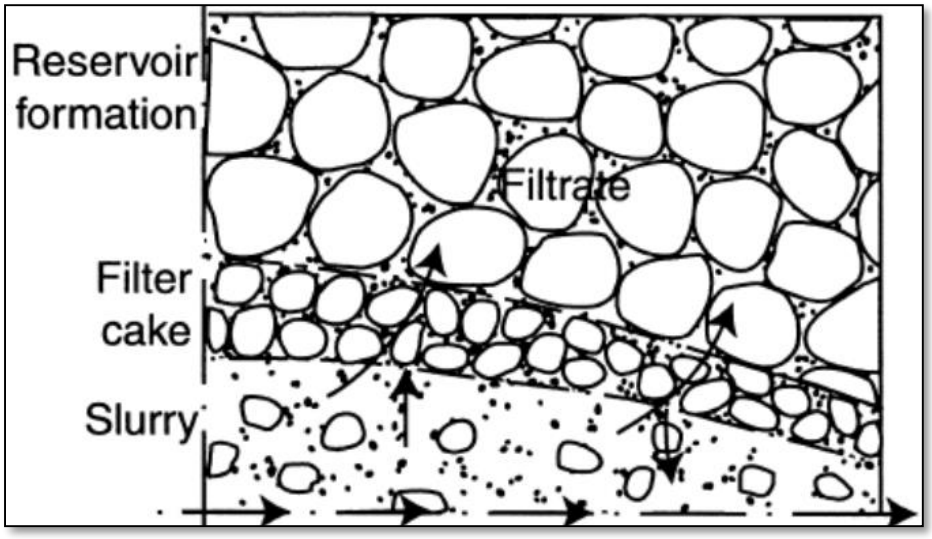

Figure 1-1: Filter cake buildup over a hydraulically created fracture surface

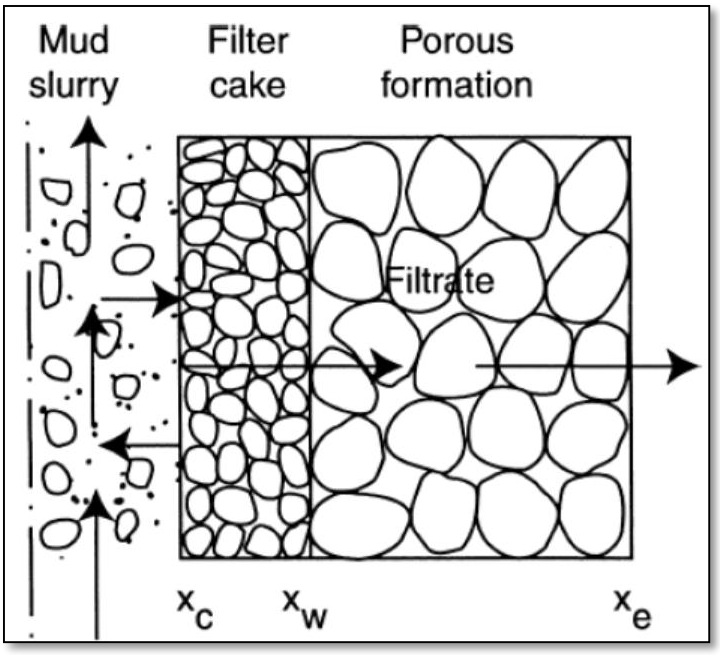

Figure 1-2: Filter cake over a flat surface of a core plug Civan (2007)

There are some factors that can control the filtration process as follow:

- Formation (permeability, porosity, pore size and structure).
- Fluid properties (cross linked or non-cross linked, power law, Newtonian or Bingham plastic, fluid additives, suspended particle size and temperature).

- Cake properties (permeability, cake compressibility, adhesive (cohesive) strength).

1.4.2. Drill-in and Completion Components

Completion fluids play an extremely important role in determining the

productivity of oil and gas wells. In a typical well work-over or completion operation, completion fluids normally stay in contact with the productive zone for one to three days. This can result in significant fluid leak off from the well to the formation resulting in potential formation damage to the near wellbore region. Maintaining a hydrostatic head in the completion fluid enough to prevent the flow of formation fluids into the wellbore is essential to control the well and prevent blowouts. The large wellbore pressure needed to offset the reservoir pressure often results in large fluid leak off, particularly when clear brines are used in high permeability formations. Large amounts of completion fluids lost to a reservoir can result in significant cost of makeup completion fluid Al-Riyamy (2000).

The drilling fluid and completion components used in drilling and completion fluids have been improving since the beginning of the oil industry. Water was the only element in the drilling fluid to clean the borehole by circulation in the drill pipe and through the

annulus. Additional studies were given to drilling fluid formulas after the beginning of horizontal drilling technology and multilateral drilling through the reservoir sections. A new type of drilling fluids was introduced for use in drilling through the pay-zone. Specially formulated drilling/completion fluids for optimizing well productivity are called drill-in fluids Xie (2001).

In addition to properties for standard drilling fluids, like providing lubrication, solids suspension, and hole stability, drilling fluid designed to protect producing intervals by

mechanically sealing the boreholes by forming thin, tough and impermeable filter cakes, maintain the wellbore stability during completion by strengthening the wellbore, and provide smooth cleanup after drilling and completion.

The main additives of the drilling fluids are bridging additives, rheology control additives, and fluid loss control additives are: -

1. Bridging Additive

   Sized calcium carbonate is the most common bridging additives used in drilling fluid. Calcium carbonate is used more widely as a weighting material because it can be dissolved in hydrogen chloride.

2. Fluid Loss Control Additive

   The drilling fluid suspended particles move with the lateral flow out of the borehole into the porous formation. The particles will be filtered near the surface and accumulate as a filter cake. This fluid loss rate is identified by the hydrodynamic forces acting on the suspended particles and determines the rate of filter cake buildup. When no small particles can be deposited are available in the suspension, the equilibrium cake thickness is achieved. Mohammed (2001). The most used fluid loss control additive is Starch.

3. Rheology Control Additive

   Xanthan is the most rheology control agent used with the drilling fluids. xanthan can build viscosity at lower concentrations than other viscosifiers. This property of xanthan provides an excellent suspending agent for cuttings and exceeds other polymer used in drilling fluids.

1.4.3. Methods for Filter Cake Removal

Filter cake removal methods including chemical and mechanical one. Mechanical methods such scraping and jetting using coiled tubing. On the other hand, efficient filter cake can be achieved chemically using breakers, are well known for filter cake removal such as live Hydrogen chloride (Hcl), organic acids, chelating and oxidizing agents, in-situ generated, enzymes or a combination of these chemicals. Todd et al. (2004) introduced emulsion inversion techniques which is mainly manipulate the physical state of the filter cake.

At the well completion stages, filter cake removal must be maximized. There are many different filter cake removal methods including chemical, mechanical and physical one. Several chemicals such as breakers, strong live acid (e.g. 15% HCL), organic acids, chelating agents, etc., can be used. Another method can be done by manipulating the physical state of the filter cake, such as by emulsion inversion. Willberg and Dismuke (2009). Outmans (1963) mentioned that the removal of particles from a filter cake managed by the frictional coefficient of the deposited particles and the filter cake itself. The drilling fluid design ability and how a given fluid build a filter cake is a must to design an optimal breaker (Hanssen 1999).

Some of the chemical methods for removing the filter cake will be discussed here

1. Emulsions

   Emulsions have been used as drilling and completion fluids. Emulsified oil-based muds are the most commonly used emulsion system in oilfield. The primary advantage of using oil external emulsions is that the formation is protected from the constituents of the brine in the wellbore. This can be a significant advantage

when working with water-sensitive formations that can cause wellbore stability problems or loss in productivity due to interactions with the wellbore water.

Adding an acid-soluble degradable oil-in-water emulsion to the appropriate completion fluid would lower the fluid loss and increase return permeability. Also, the amount of solids needed to form a filter cake would be reduced depending on the oil percentage and the particle size distribution. Consequently, these low solids, low filtrate systems deposited less filter cake providing maximum removability in open hole completions. Emulsions are also good lubricants and help in freeing stuck pipes Al-Riyamy (2000).

2. Enzymes as Clean-up Chemicals

Enzymes are potentially very powerful clean-up chemicals because they are active at mild conditions of temperature and near neutral pH, high specificity, catalytic efficiency, and excellent HSE profile. The first field application of enzymes was to remove fracturing fluid residues. This method is excellent option to use filter cake removal due to high efficiency cleanup, large cost savings and avoiding the need for acid, Hanssen et al. (1999). An enzyme normally accelerates the reaction as the temperature is increased, Hanssen et al. (1999). Enzyme breaker system provides efficient filter cake removal with less contact time, Rickards et al (1993). Enzyme can remove the fitter cake formed by calcium carbonate easily, Price-Smith & Bennett, (1996). Mathew & Goerge w, (2007) concluded that enzyme with stabilizer is suitable for high temperature (250°F) areas.

3. Acidic Completion Fluids

Acidic brines can be used in the field to remove the filter cake but is not recommended to be used in horizontal or maximum reservoir contact (MRC) wells due to high reaction rate of the acid with the formation, especially at high temperatures and high volume required. The high reaction rate prevents effective filter cake removal such wells. Al-Yami and Nasr-El-Din (2009). Siddiqui and Nasr-El-Din (2005) concluded in their sandstone core flooding samples that acidic brine (pH=4) is not effective in removing drilling fluid filter cake.

4. In-Situ Generated Acids

This system of in-situ generated acid technology to remove formation damage and minimize conventional breakers risk, it has special chemical components, and designed to dissolve some solid particles present in the filter cake by generating organic acid in situ. It provides a delay in reaction, distribute regularly among the horizontal section, and therefore, minimize the risk of corrosion, Binmoqbil et al. (2009).

Nasr-El-Din & AL-Otaibi, (2005) concluded their study of the effect of using in-situ generated acids, which created acetic acid, for cake removal; in-situ generated acid can eliminate most of the acid-soluble material only. The temperature window for in-situ generated acids is no higher than (200°F).

AlMoajil et al (2014) reported that a combination of low concentration of strong acid such as Hcl with an effective organic acid can be effective in removing the filter cake that created by manganese tetroxide. AlMoajil A et al (2008) reported that the organic acids rate of reaction with manganese oxides controlled by the acid structure, the reactants stoichiometry, and the capability of the chelate function in the acid.

Brady & Bradbury, (2000) mentioned the oxidizing agent are widely used in the energy industry for filter cake removal. Albonico et al (2007) reported that the most common oxidizing agent is the potassium persulfate which is very effective in low temperate range. Also, introduced new oxidizing agent that is effective at temperature lower than 50C and desired delay time can be obtained.

5. Chelating Agents

Elkatatny (2013) reported that calcium carbonate filter cake can be efficiently removed using chelating agents. The solution of 20 wt.% of GLDA and 20wt.% of HEDTA was used when using Polylactic acid as a component of the drilling fluid. He also showed no formation damage was observed when using chelating agents as a breaker to remove the calcium carbonate filter cake. Almubarek T et al (2017) showed that chelating agents such EDT HEDTA, DTPA, etc are very common application in cake removal. He reported that chelating agents are often used in high temperature and show good dissolving power, low corrosion, low slugging tendencies.

Mathew & Goerge w, (2007) showed that a chelating-enzyme is very effective for cleaning up the open hole mud, gravel pack completions and have good returned permeability result. (Burton et al. 2000). Fig. 3.9 shows the molecular structure of ethylenediaminetetraacetic (EDTA). Elkatatny et al. (2013) showed that ilmenite filter cake cannot be removed completely by some chelating agents. HEDTA of 20wt.% dissolved only 31 wt.% of iron and 20wt.% of titanium. In addition to that, EDTA dissolve only 39 wt.% of iron and 25wt.% of titanium after 16 hours of soaking. Elkatatny et al. (2018) reported that barite-removal efficiency is 87% when used either low pH chelating agents or EDTA at ph of 12. This can be done after converting barite to barium carbonate (BacCo3) using a combination of potassium hydroxide (KOH) and potassium carbonate (K2Co3).

CHAPTER 2
LITERATURE REVIEW

2.1. Weighting Materials in Drilling Fluids; Advantages and Disadvantages

Haaland et al. (1976) reported weighting materials are components that are usually added to the drilling fluids dissolved or suspended to provide a homogeneous fluid column (control density) that can help fluids to perform all kinds of drilling fluid functions. Common weighting materials utilized in drilling are hematite, bentonite, manganese tetroxide, barite, and calcium carbonate. Bageri et al (2013) conclude that more than 70% of the mineral composition of filter cake can be attributed to the drilling fluid weighting material. Each one of these materials has its own advantages and limitations.

For many years, barite ($BaSO_4$) has been the most used weighting material in drilling fluid. Barite quantity is available with huge amount in the market. The heavy metal components associated with barite such as cadmium, mercury, arsenic, and lead are the main source of pollution.

On the other hand, Haaland et al.(1976) reported that barite can cause a formation damage due to insolubility in Hcl which leads to inefficient filter cake removal.

Rae et al. (2001) reported that barite also produces additional frictional pressure that leads to extreme equivalent circulating density (ECD) and increase the formation damage degree due to insoluble in most solvent. Paswan et al. (2016) reported that barite sag is a very substantial variation in drilling fluid density created by the settlement of barite in high-

angle wells. It also can be minimized by better well planning, drilling fluid properties, and excellent operation flow up Bern et al. (1996).

Suri (2005) reported that calcium carbonate is known as adequate bridging additives used in drilling muds. Calcium carbonate can substitute the barite function in oil-based mud due to its faster dissolution in oil than barite. Carbonate particles has other main advantages to be used in drilling fluids such as acid solubility for filter cake dissolution before production and particle size variations that reduce the formation invasion. However, the calcium carbonate specific gravity range is 2.6-2.8 which lead to a limitation of the drilling fluid maximum density to a 12 lb./gal. This density limitation is a disadvantage of calcium carbonate in HPHT conditions.

ALMoajil A (2010) reported manganese tetraoxide (Mn_3O_4) has unique specifications to create high density drilling fluid with low solid content and settling such as high specific gravity of 4.8 g/ cm3, spherical in shape, and small particle size reach up to 1 μm. Kleverlaan & Lawless (2004) stated that manganese tetroxide used in drilling fluids to reduce the mud rheology and sag issue at lower cost. AlMoajil et al (2014) reported that organic acids are not effective with high-density drilling fluids that have huge quantities of weighting materials such as manganese tetroxide.

Another important weighting material used in drilling fluid is Hematite. Hematite is an iron oxide (Fe_2O_3). Morgenthaler et al., 2000 reported hematite can increase the drilling fluid density to 17 lb./gal, however it will produce a large quantity of iron if removed by Hcl.

(Howard, 1995) indicated that hematite is more abrasive and has high tendency of sag. In addition to that, hematite large particles may cause severe slumping at long static drilling situations, Bern et al. (1998).

2.2. Ilmenite Evolution

The evolution of utilizing ilmenite as weighting agent in the drilling fluids is introduced to the energy industry on 1979. It was used to avoid shortage, high cost, of barite, Blomberg (1984) and Rae et al. (2001) .

Idris et al. (1994) reported in his study on the Malaysian ilmenite as a weighting material in drilling fluid that ilmenite has the potential to be utilized as weighting agent in drilling fluid. It showed ilmenite gave a lower solid content compare to barite due to high specific gravity of ilmenite. He also observed higher yield point and gel strength even thought this issue can be reduced by increasing the lignosulfonate concentration.

Ismail et al (1999) showed that ilmenite could produce the same mud weight as barite with smaller quantity, which yields lower solids content. the ilmenite has lower fluid loss than barite which lead to formation damage reduction. According to Fjogstad et al (2000) there are no disadvantages have been seen in using ilmenite in the drilling fluid. Ilmenite is suited to be circulated and used again because it has a lower tendency to be groaned down to finer materials and therefore was less of a need for fluid dilution.

The experiment work did show the performance of ilmenite is equal or even better in some cases than barites in water-based drilling fluid.

Saasen et al. (1993) reported the ilmenite advantages of using a 10 μm grade such as high abrasion degree caused by high concentration of coarse grains, the drilling fluids have a

longer life time, and a reduced need for dilution when using ilmenite because of the good stability of the fluid.

Amighi & Shahbazi (2010) reported ilmenite has better quality from environmental prospective than barite. In water-based drilling fluid, it eliminates the heavy metals discharge, it also shows less sag than barite in HPHT deviated drilling. The ilmenite erosion affect can be reduced to a level less than barite by adjusting the particle size distribution of ilmenite particularly when mean size become around 10 microns. They also reported no dynamic sag was seen at drilling time or running casing and liner while using ilmenite as a weighing material in drilling fluids.

Al-bagoury & Steele (2012) emphasize the importance of some ilmenite features that can be implemented in drilling operation especially horizontal drilling, slimehole, and deep wells. These are the micronized ilmenite with an average size (D50) of 5 μm is potential drilling fluid agent that showed low sag tendency, and low plastic viscosity compare with barite at same conditions.

Elkatatny et al. (2012) studied the ilmenite as drilling fluid weighting material in HPHT conditions. Ilmenite showed good stability when mixed with water at a PH above 7 and was dispersed and stable when mixed with drilling fluid. No sag problem was observed. The filter cake thickness is 0.2 in and 12 cm3 filtrate volume under dynamic condition. The ilmenite filter cake was heterogeneous and has ilmenite particles especially closer to rock surface.

Al-bagoury (2014) introduced a new micronized ilmenite grade (5 µm) in drilling fluids. it shows high density 4.6-4.7SG, high hardness (Mohs nr. 6), low heavy metal content, excellent dynamic sag, stable rheology, less viscosity than barite, dynamic filtrate was in accepted rate (<5 cm3) and very cost-effective.

2.3. Ilmenite Reaction and Removal

The ilmenite chemical reaction can be defined by the following chemical reaction:

$$FeTiO3 + 2HCl \longrightarrow Fe2^{+} + TiO2 + 2Cl^{-} + 2OH^{-}$$

, Van Dyk et al (2002).

Jackson & Wadsworth (1976) conclude that the ilmenite dissolution rate does not depend on the particle size. He tested different size fractions in same conduction where iron and titanium extracted within 250 min for all fractions. However, Olanipekun (1999) showed that particle size has no effect on the ilmenite dissolution rate. The rate of iron and titanium extraction has reverse relation with the particle size. Jackson & Wadsworth also showed the rate of ilmenite dissolution is strongly controlled by the acid concentration.

Husseinet al. (1976), showed mainly iron was dissolved, while Jackson & Wadsworth (1976) showed both titanium and iron are dissolved. Sinha (1984) showed the rate of ilmenite dissolution increases quickly with an increase in leaching temperature. Tsuchida et al. (1982) and Sinha (1984) showed no major change in the iron or titanium extraction rate with in an increase in stirring speed in a range of 100–500 min.

Jackson and Wadsworth (1976) showed the initial acid-to-ilmenite mole ratio whether both iron and titanium goes into solution or whether only iron goes into solution.

Duncan & Metson (1982) reported that the dissolution rate of both iron and titanium increased when bisulphate and fluoride added to the leach solutions. In addition to that, Girgin (1990) showed that the iron and titanium dissolution rate increased intensely when added more methanol. Abdel-Aal & Ibrahim (2000) showed the leaching mechanism of ilmenite with sulfate is slow, expensive and the ferrous sulfate is environmentally unsafe.

Elkatatny et al. (2013) introduced using the hydrogen chloride to remove the ilmenite filter cake in water-based. HCL (10 we%) showed good dissolution about 75 wt.% of the iron form ilmenite particles after 10 hours at a temperature of 300 °F. it is also illustrated that the removal of ilmenite water-based cake procedure is simpler comparing with barite water-based filter cake.

CHAPTER 3

EXPERIMENTAL METHODOLOGY AND SET UP

3.1. Ilmenite Kinetic Reaction Experiment

- Materials
- A sample of Ilmenite with an average particle size of 5 microns was utilized in this research. The TURBOTRAC Particle Size Analyzer shown in Figure 3-1 was used to show the ilmenite particle size distribution that shown in Figure 3-2 Ilmenite samples were also categorized using scanning electron microscopy, SEM, Figure 3-3 to evaluate the ilmenite elemental composition of ilmenite are shown on Figure 3-4, Figure 3-5 and Table 3-1. The received ilmenite sample was confirmed very clean without any contamination as shown in Figure 3-6: ilmenite Purity SEM result using X-Ray diffraction (XRD) techniques shown in Figure 3-7: XRD Machine, D8 Advance. DTPA, GLDA, EDTA chelating agents of initial concentration of 40 wt.% were used. The chelating agents was diluted with deionized water to obtain the desired weight concentration of each experiments. GBRF have used in different concentration. On the other hand, Potassium carbonate, potassium hydrogen carbonate, and potassium chloride have been tested as catalysts to evaluate the ilmenite dissolution rate. The pH of each experiment solution has been adjusted using NaOH and Hcl.

Figure 3-1:TURBOTRAC Particle Size Analyzer

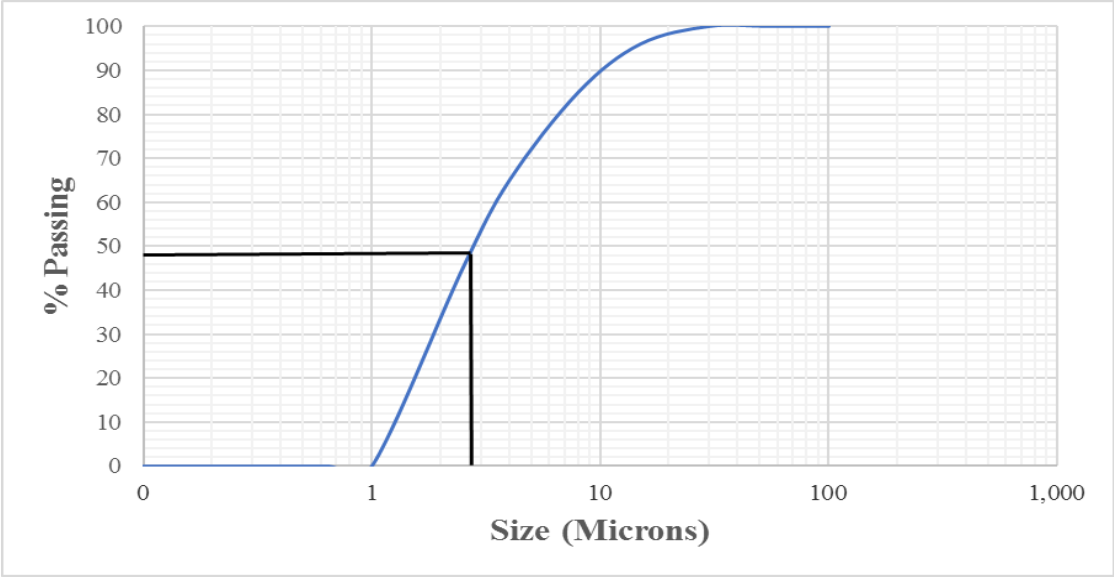

Figure 3-2: Particle Size Distribution of Raw Ilmenite Sample

Figure 3-3: SEM Machine

Table 3-1: Elemental Composition of Raw Barite Sample

Elements	Wt.%
O	31.4
Fe	30.1
Ti	22.5
C	9.9
Cu	2.8
Mg	1.8
Si	1
AL	0.3

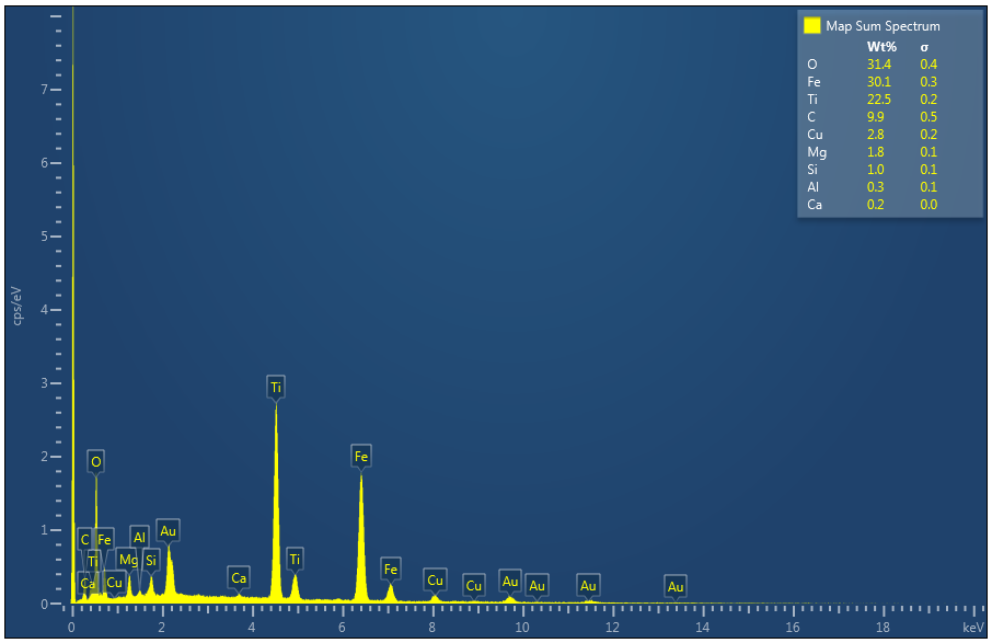

Figure 3-4: Elemental Composition of Raw Ilmenite Sample SEM

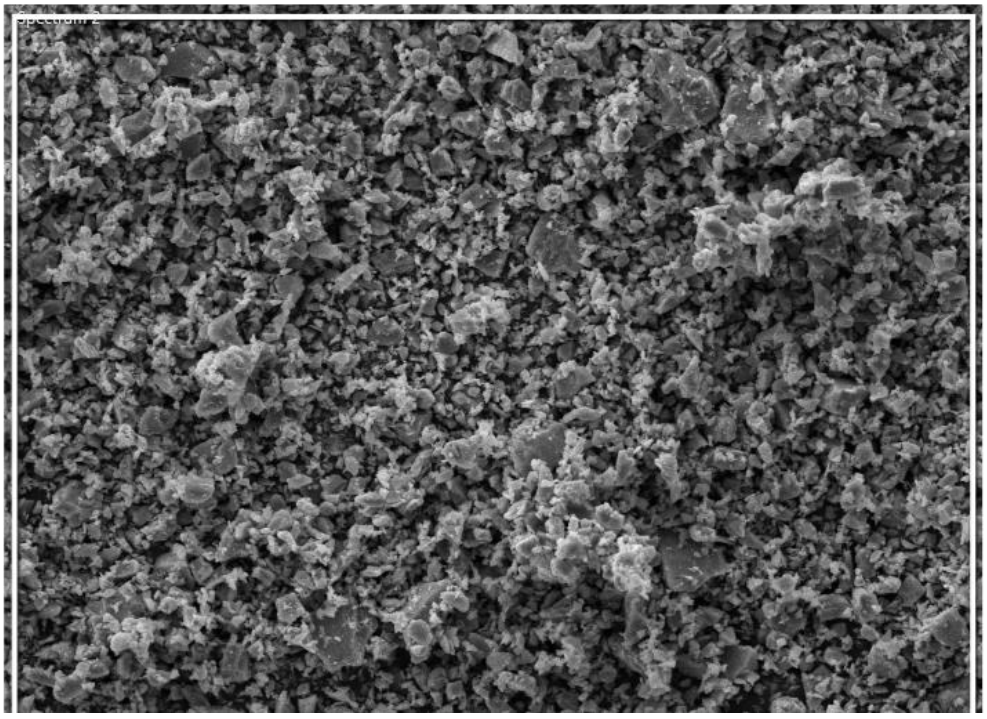

Figure 3-5: SEM micrographs of Ilmenite as received

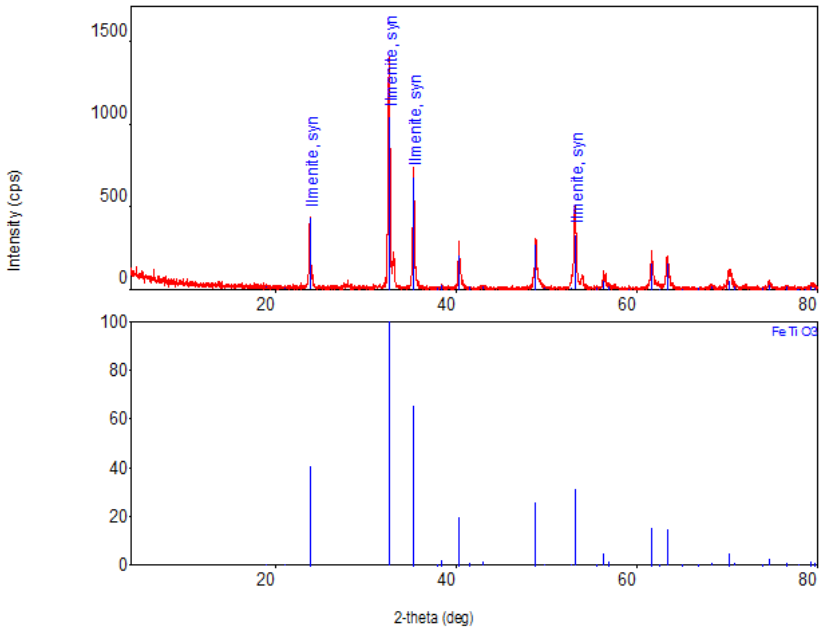

Figure 3-6: ilmenite Purity SEM result

Figure 3-7: XRD Machine, D8 Advance

- Procedure

  In this experiment, ilmenite solubility tests were performed to quantify the amount of ilmenite particles that can be dissolved. The reactions were conducted for several times with different chemical combination conditions. Table 3-1show the summary of the solubility test parameters. The deionized water was used in some experiment to dilute the chemical weight percentages. The pH was measured using pH meter showed in Figure 3-8. Ilmenite were added to the solution, with a ratio of 1 g to 50 ml, and put on a multipole heating magnetic stirrer for 24 hours at a temperature of 300 °F at dynamic condition. A condenser was used to assure no fluid evaporation. Figure 3-9 shows the setup of the solubility test. The test duration is 24 hours, then, the filtration setup shown in Figure 3-10 was used to filter the sample using filter paper to measure the ilmenite solubility. After that, the dry process on the filtered solid conducted for 2-3 hours at the oven shown in Figure 3-11. The filter paper weight was measured before and after the filtration test using weight balance (accuracy: 0.0001 g) shown in Figure 3-12. Then, the solubility was calculated in wt.% using equation (1).

$$\text{Solubility}, \% = \frac{W_{Solids} - (W_{after} - W_{before})}{W_{Solids}} \text{x}100 \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

  where;

  Wsolids= ilmenite solid particles weight, g

  Wafter= filter paper weight after the test, g

  Wbefore= filter paper weight before the test,

\

Table 3-2: Solubility Test Summary

Parameter	Description
Ilmenite	1 gram
Total Solution	50 mL
Chemicals (DTPA, GLDA, EDTA, HCL .)	10,15,20… wt.%
Temperature	300 °F
Condition	Dynamic
Solubility Duration	24 hours
Drying Duration	2-3 hours

Figure 3-8: JENWAY 3510 pH Meter

Figure 3-9: Solubility Test Setup

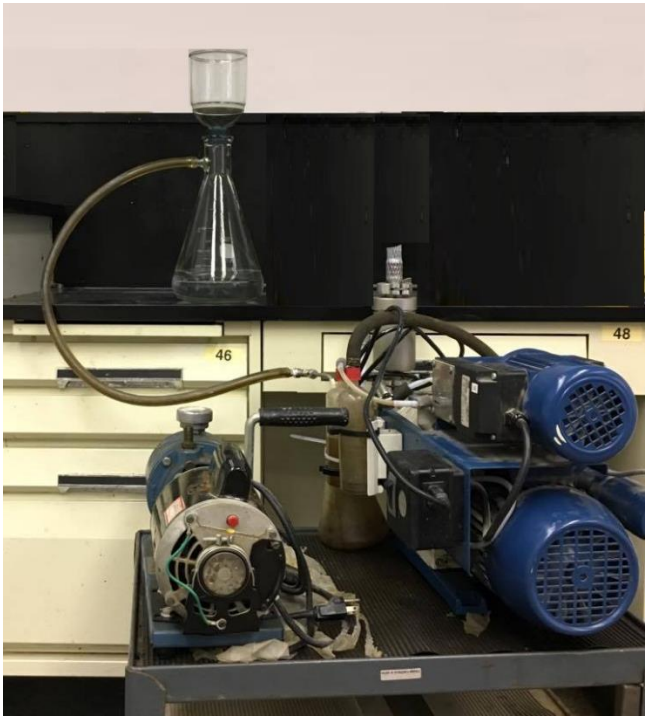

Figure 3-10: Filtration Setup

Figure 3-11: The Roller Oven

Figure 3-12: Weight Balance

3.2. Fluid Preparation and Rheology Measurements

Water-based drilling completion contained ilmenite (300 g) as a weighting material with a density of 98.6 pcf. was prepared using drilling fluid mixer Figure 3-13. Then, Defoamer was added to avoid the foam formation. The other additives were added gradually for

mixing as follows, xanthan gum (1 g) to control viscosity, modified starch (5 g) for fluid loss control, PAC-R for fluid loss control at high temperature, potassium chloride (72 g) for controlling the shale inhibition, potassium hydroxide (1 g) for pH control, fine and medium (14 g) calcium carbonate as bridging material respectively. The drilling fluid was mixed to form 480 cm^3.The rotational speed was gradually increased from low to high as viscosity built up. Table 3-3 indicate all the additives, their ratios and functions, and the mixing time. After that, the fluid rheological properties have been measured in high and low temperature using mud balance, Fan 35A Viscometer, HPHT Rheometer, shown in Figure 3-14, Figure 3-15, and Figure 3-16 respectively.

Table 3-3: Ilmenite Drilling Fluid Formulation

Name	Lab Unit, g	Mixing Time, min	Purpose
Water	290	-	Base
Defoamer	0.08	-	Anti-foam
Xanthan gum	1	10	Viscosifier
Modified starch	5	10	Fluid Loss
PAC-R	1	10	Fluid loss/ Filtration control
KCL	72	20	Density and shale inhabitation
KOH	1	10	pH Adjustment
CaCO3 Fine (25 um)	7	10	Bridging Agent
CaCO3 Medium (50 um)	7	10	
Ilmenite	300	10	Weighting Material

Figure 3-13: Drilling Fluid Mixer

Figure 3-14: Drilling Mud Balance

Figure 3-15: Fan 35A Viscometer

Figure 3-16: HPHT Rheometer

3.3. HPHT Filtration Test

The drilling fluid formulation was evaluated using HPHT filtration tests to measure the fluid loss into the formation. A HPHT filter press was used to conduct the filtration procedure. An Indiana limestone core of 1 in thick and 2.5 in diameter were used in the test. The filtration test conducted at 250˚F and 300 psi differential pressure at static condition. The filtration was performed using the same drilling mud for different filtration time periods. the filtration test was performed at various times from 1 to 30 min. Before the filtration process, the limestone core was fully saturated by fresh water using special set-up (Filter-Press with continuous pumping). The core sample was weighted before and after saturation. Table 3-4 show the HPHT filtration test parameters.

Then, the following process was applied:

1. The 450 ml of drilling fluid was poured in the HPHT filter cell and the temperature was modified at 250°F and the pressure at 300 psi for 25 min.
2. The bottom valve of the HPHT cell was gradually opened, and the filtrate volume was collected for the determined time.

Table 3-4: HPHT Filtration Test Summary

Parameter	Description
Core thick	1 in
Core Diameter	2.5 in
Core Average Permeability	20 md
Core Average Porosity	12 %
Pressure	300 psi
Temperature	250 °F
Condition	static
Drilling Fluid volume	450 ml
Filtration Duration	30 minutes

3. The bottom valve was closed, and the HPHT cell was left for cooling for 40 minutes.
4. The core sample with filter cake was removed from the cell.
5. The core sample weight, and thickness were measured.
6. Repeat the same steps using for all the core samples the time.

Figure 3-17: HPHT Filter press, 500 ml, OFITE

3.4. Filter Cake Removal Test

The HPHT filter press apparatus was used again to evaluate the effectiveness of optimized solution: 300 ml of GBRF (75 wt.%), corrosion inhibitor (2 wt.%), Intensifier (2 wt.%), and DI (21 wt.%) in removing the filter cake formed on Indiana limestone core sample by ilmenite drilling fluids. The test was conducted for 24 hours under static condition at 250°F and 300 psi. Eventually, the core sample weight and thickness were measured after removal to able to quantify the filter cake efficiency. Table 3-5 showed the summary of filter cake removal test.

Table 3-5: Filter Cake Removal Test Summary

Parameter	Description
Core thick	1 in
Core Diameter	2.5 in
Core Average Permeability	20 md
Core Average Porosity	12 %
Pressure	300 psi
Temperature	250 °F
Condition	static
Optimized Solution	300 ml
Duration	24 hours

3.5. Permeability Experiment and Calculation

The permeability of Indiana limestone core sample was calculated before filtration and after the removal test. The flow rate and differently pressure was obtained to apply Darcy's law on each core to calculate the initial permeability. The final permeability was obtained by the same procedure after the removal. This experiment will measure the efficiency of the new removal formula. Filter cake removal efficiency was calculated using equations: (2) & (3). In addition to that, equation (4) is used to calculate the retained permeability.

$$\text{Removal Efficiency}, \% = \frac{W_{Filter\ cake} - (W_{d3} - W_{d1})}{W_{\text{Filter cake}}} \text{x} 100 \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

$$W_{\text{Filter cake}} = (W_{d2} - W_{d1}) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where;

$W_{\text{Filter cake}}$= filter cake weight, g

W_{d1}= saturated ceramic disc weight, g

W_{d2}= ceramic disc after filtration weight, g

W_{d3}= ceramic disc after removal weight, g

$$\text{Retained Permeability, \%} = \frac{K_f}{K_i} x100 \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

K_f = Final Permeability, mD

K_i = inital Permeability, mD

CHAPTER 4

Results and Discussion

4.1. Ilmenite Kinetic Reaction Result

Solubility test was conducted to evaluate the different chemicals effect on ilmenite dissolution. Figure 4-1, Figure 4-2, Figure 4-3, respectively show the results of solubility test of ilmenite in DTPA, EDTA, and GLDA at different weight concentrations in both high and low pH. In general, the ilmenite dissolution rate was very low with no change in color of the black color of the solution. 27% of ilmenite dissolution rate was recorded as the maximum when ilmenite dissolved by 20 wt.% of GLDA at low pH of 4. Also, the solution composition changes slightly trying to enhance the ilmenite dissolution rate. Figure 4-4 show the result of ilmenite in 20 wt.% DTPA of pH:5 at different GLDA concentrations (10 – 15 – 20) wt.%. there is a slight increase in ilmenite dissolution to 34% at 15wt.% of GLDA. On the other hand, Figure 4-5, Figure 4-6, and Figure 4-7, show the impact of adding Hcl to chelating agents where there is no improvement in ilmenite dissolution rate accept in using 1wt.% of Hcl in 15wt.% of GLDA. In addition to that, some chemicals such as (Potassium carbonate, potassium hydrogen carbonate, and potassium chloride) were added to the solution as catalyst where there is no enhancement in the ilmenite dissolution rate, but it also had a negative impact such as potassium chloride in Figure 4-8.

GBRF has shown good result when used to dissolve ilmenite. It shows increasing trend when used form 25 wt.% of the solution to 100 wt.% as shown in Figure 4-9. GBRFI repeatability test shows effective ilmenite dissolution rate of 82% after 24 hours' period in Figure 4-10. Figure 4-11, show the effect of time to the ilmenite solubility using 100wt.%

where ilmenite solubility at 8 hours showed the best time of solution to dissolve the ilmenite. In addition to that, there is no remaining ilmenite particles after the reaction and the solution color was completely change to yellow, Figure 4-12. Figure 4-13 showed there is slightly reduction of ilmenite solubility when the temperature reduced to 250 ^{o}F.

Figure 4-14, showed the effective GBRF formula that is both effectively can dissolve ilmenite and not corrosive. The solution is 75wt% of GBRF, 2wt.% corrosion inhibitor, 2 wt.% intensifier. This system will be used to remove filter cake that will be filtered on a core sample.

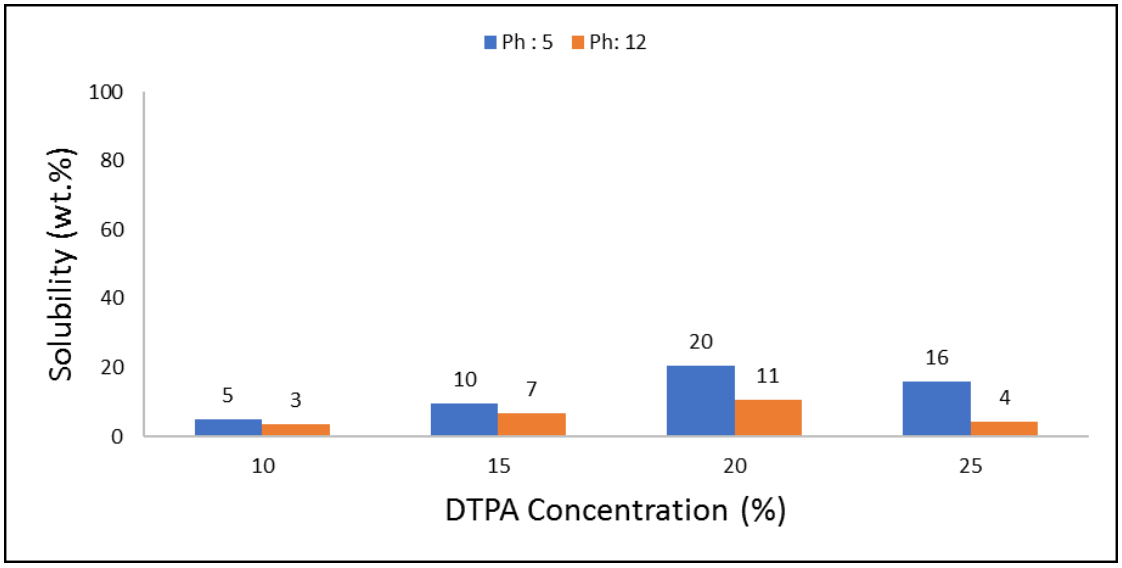

Figure 4-1: The Ilmenite Solubility in DTPA.

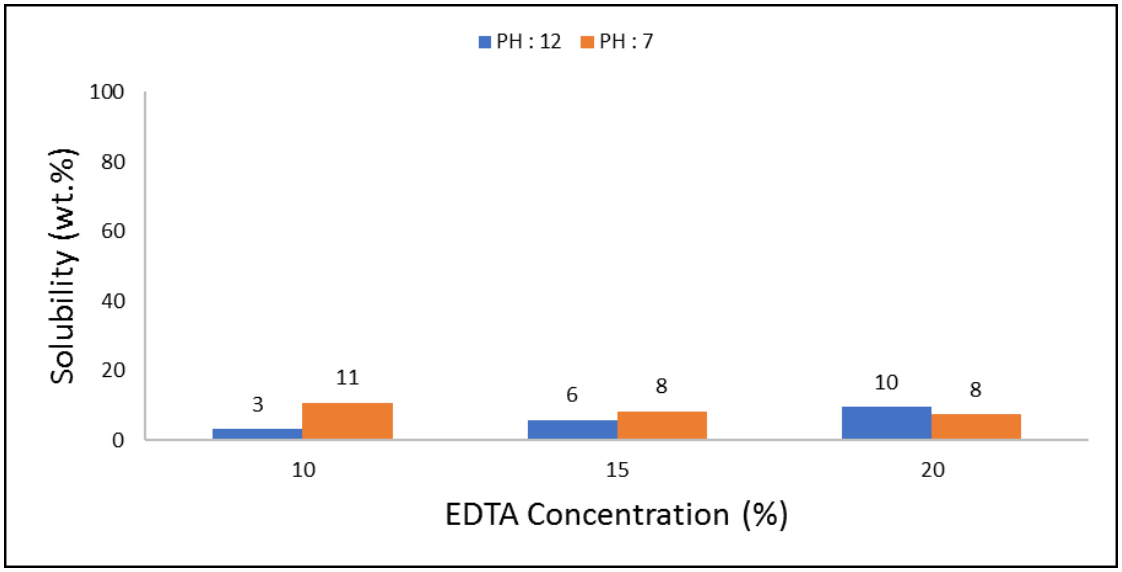

Figure 4-2: The Ilmenite Solubility in EDTA.

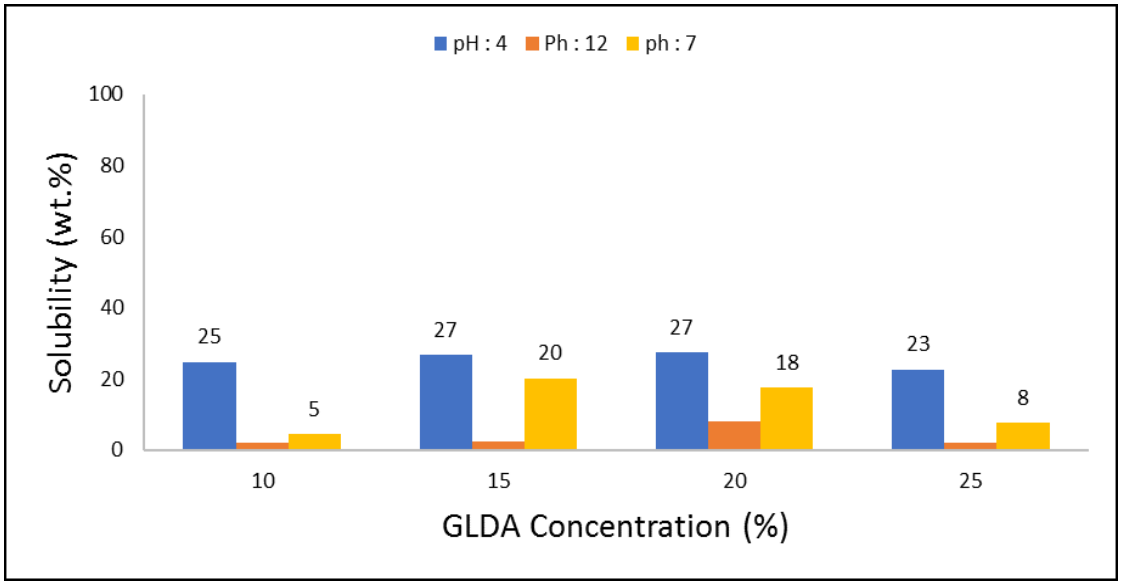

Figure 4-3: The Ilmenite Solubility in GLDA.

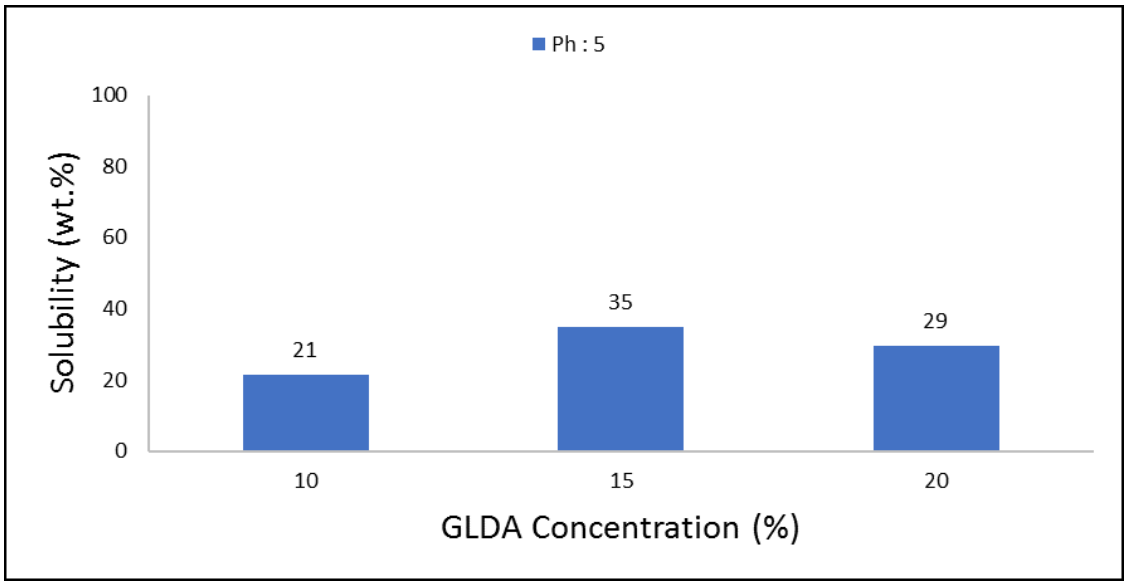

Figure 4-4: The Ilmenite Solubility in 20 wt.% DTPA at different GLDA wt.%.

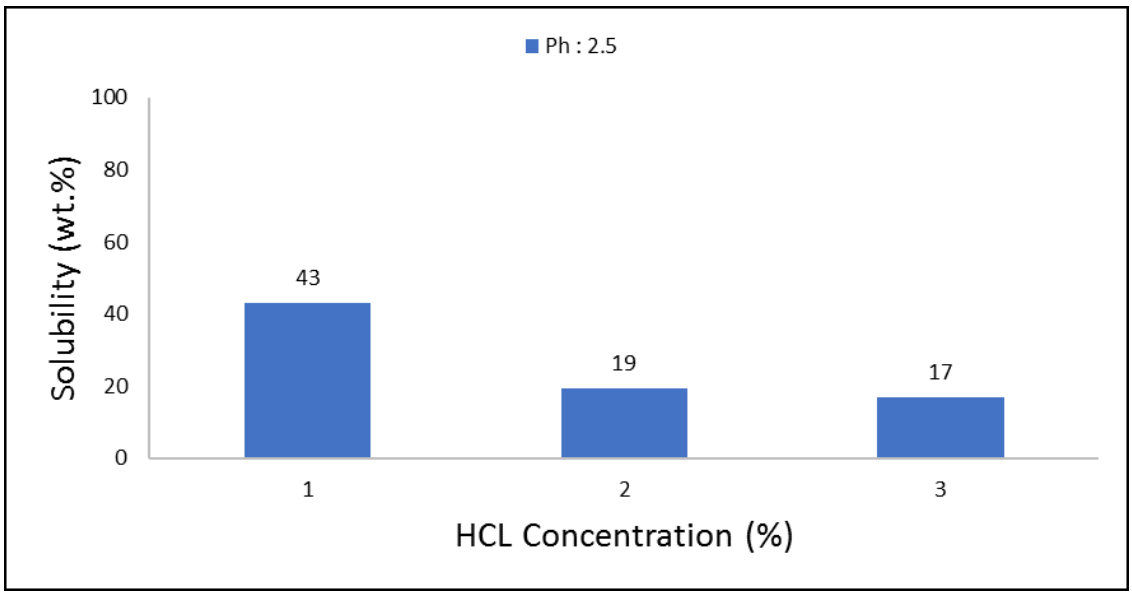

Figure 4-5: The Ilmenite Solubility in 15 wt.% GLDA at different HCL wt.%.

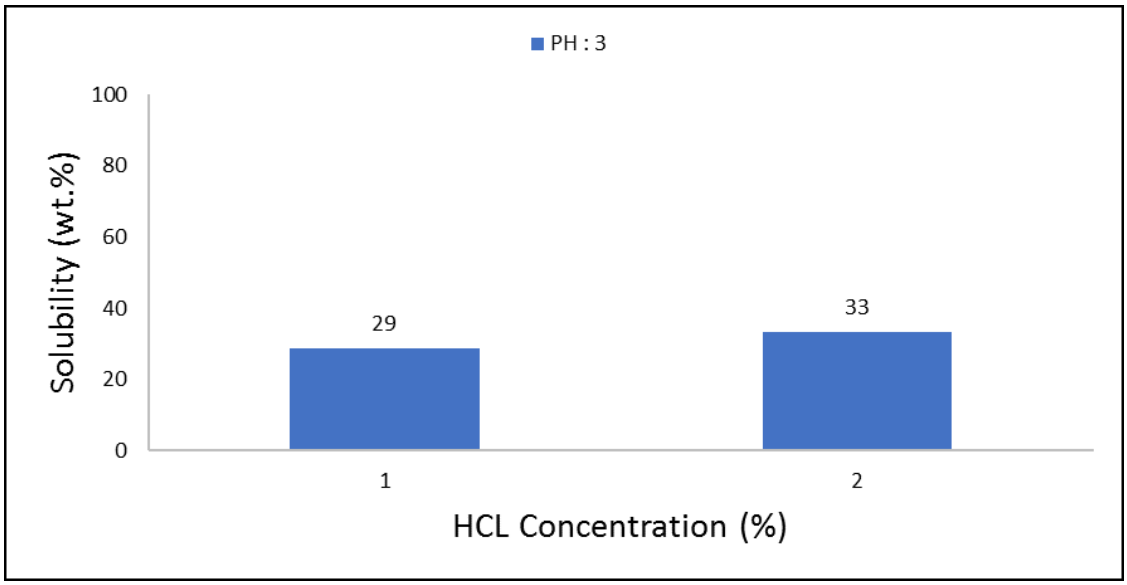

Figure 4-6: The Ilmenite Solubility in 20 wt.% DTPA at different HCL wt.%.

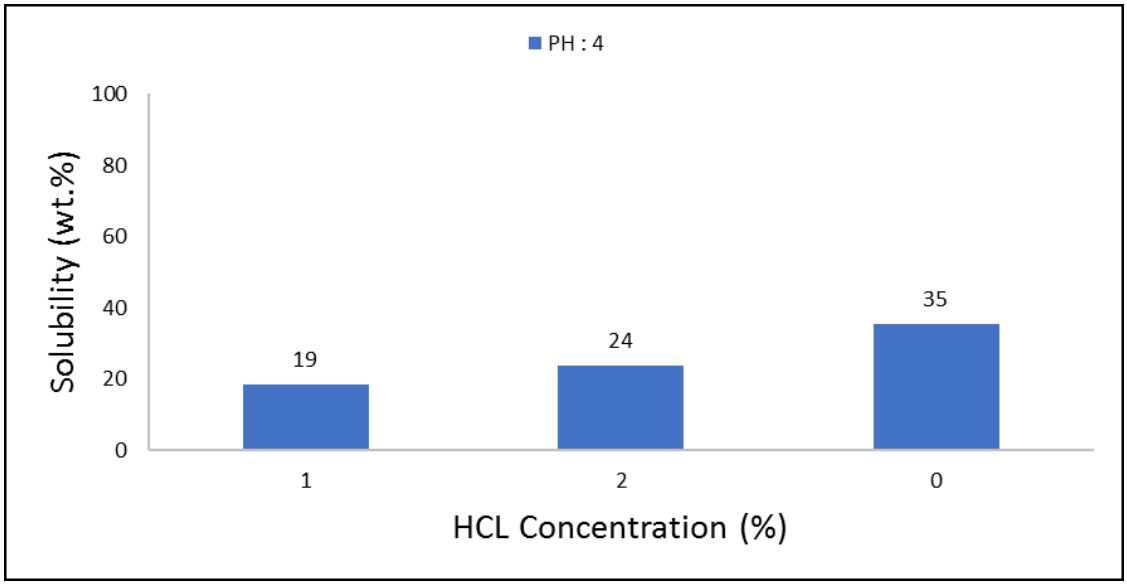

Figure 4-7: The Ilmenite Solubility in 20 wt.% DTPA & 15 wt.% GLDA at different HCL wt.%.

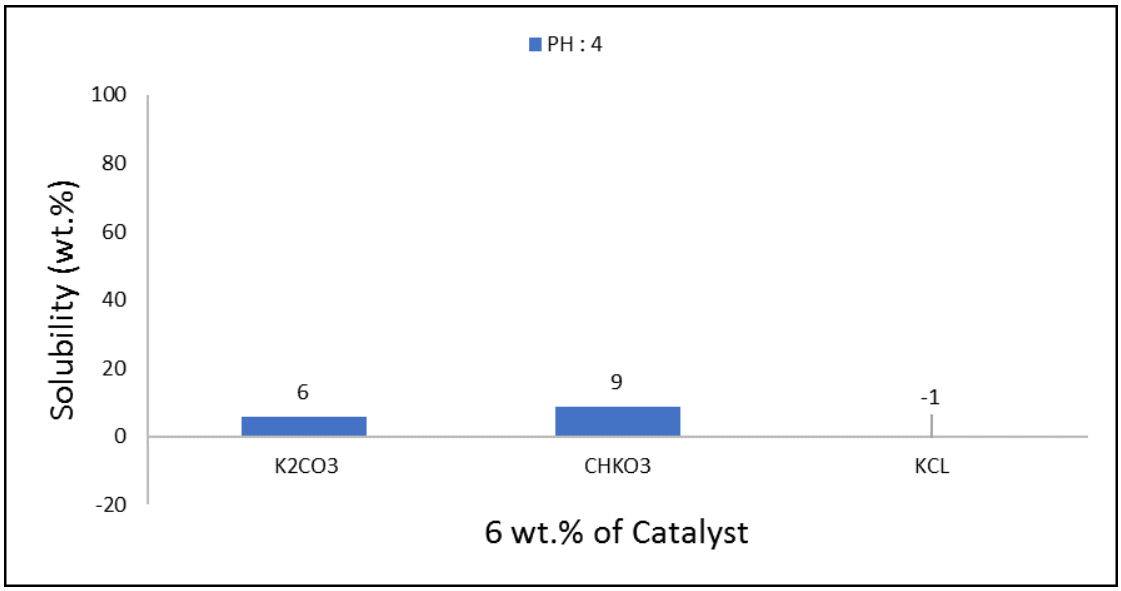

Figure 4-8: The Ilmenite Solubility in 20 wt.% DTPA & 15 wt.% GLDA at 6 wt.% of different Catalysts.

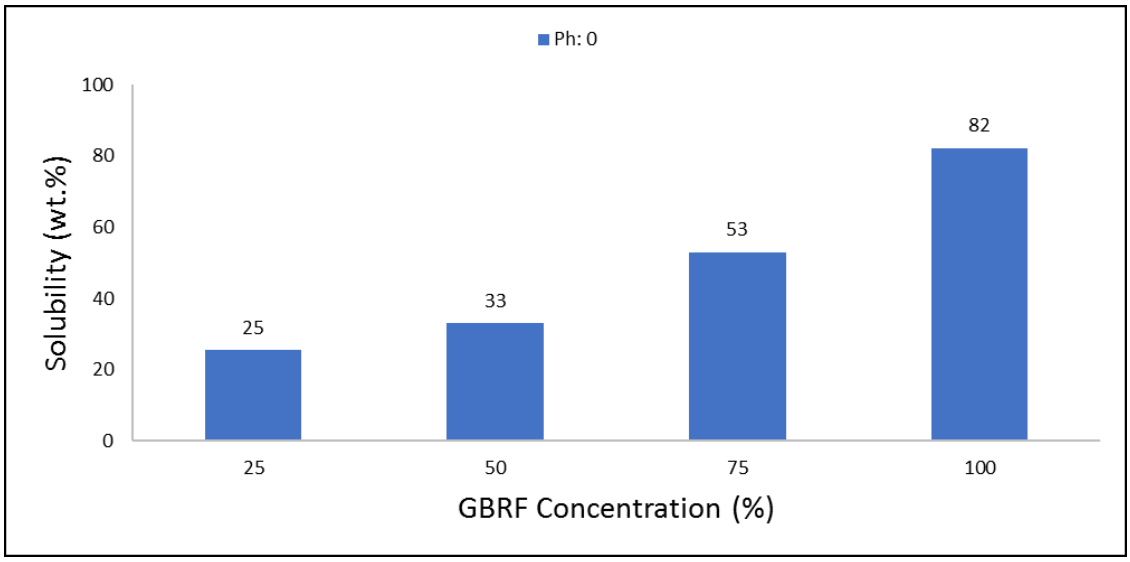

Figure 4-9: The Ilmenite Solubility in 100 wt.% GBRF.

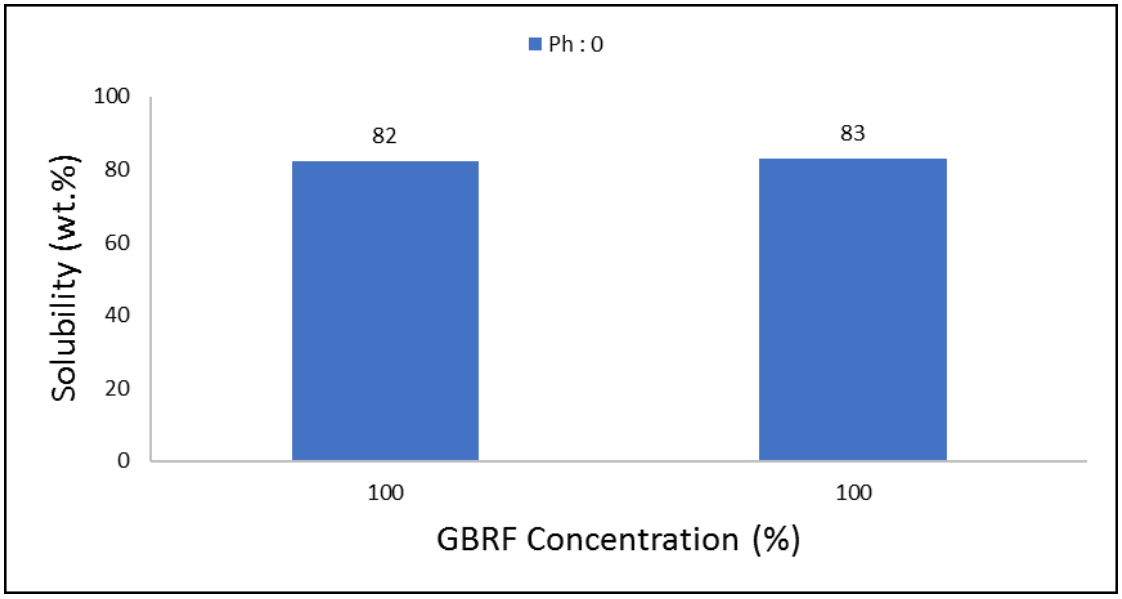

Figure 4-10: The Ilmenite Solubility in 100 wt.% GBRF (Repeatability Test).

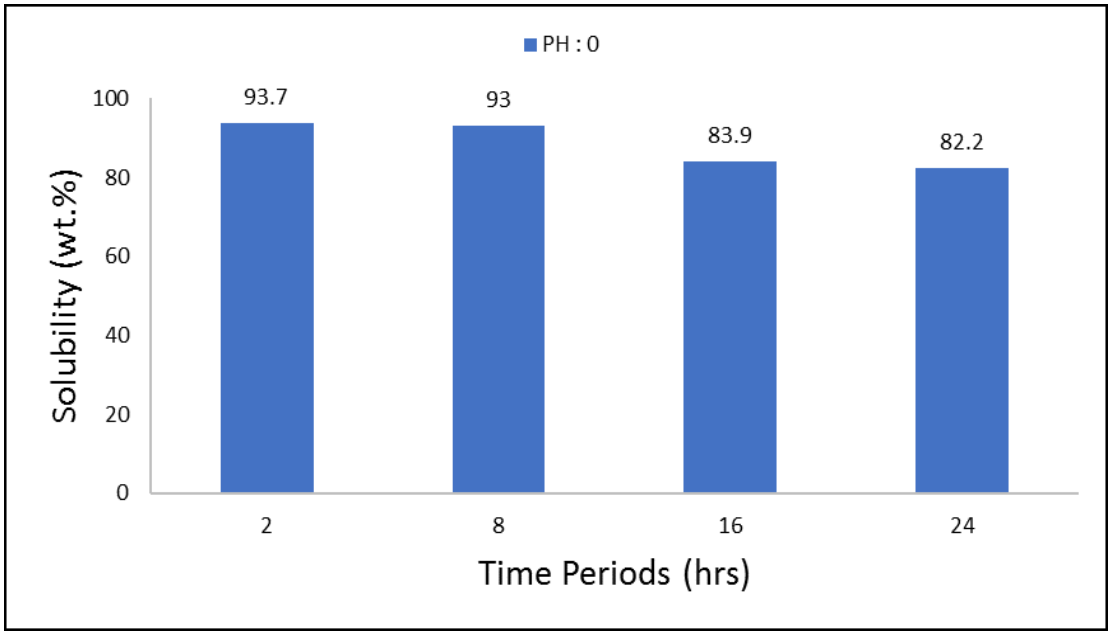

Figure 4-11: The effect of time on Ilmenite Solubility in 100 wt.% GBRF.

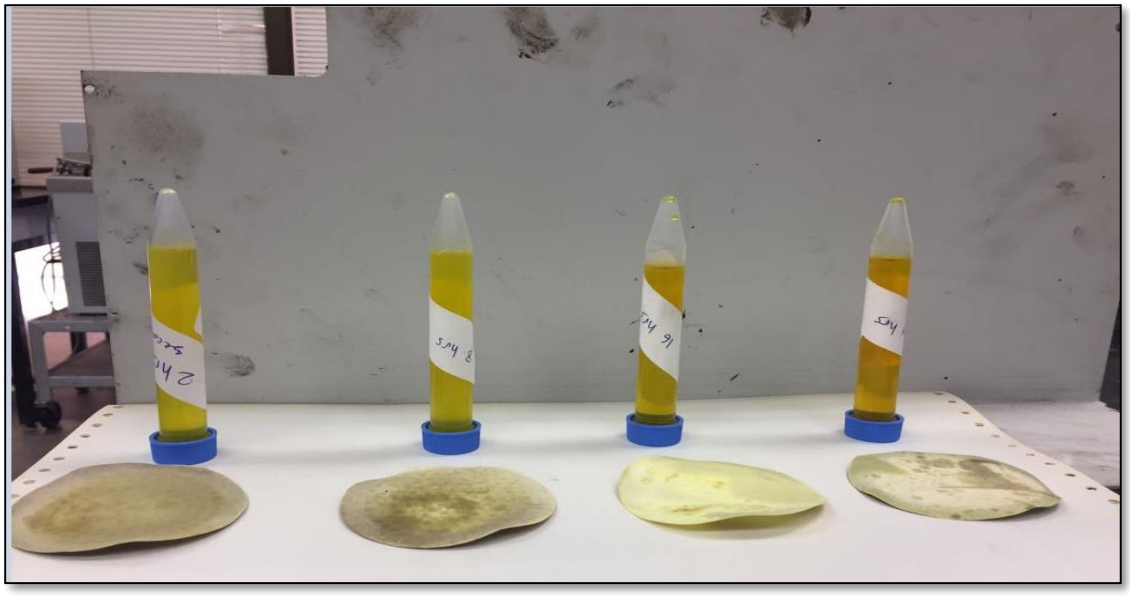

Figure 4-12: A) Filtrated solution after the reaction, B) Remaining solid after the reaction.

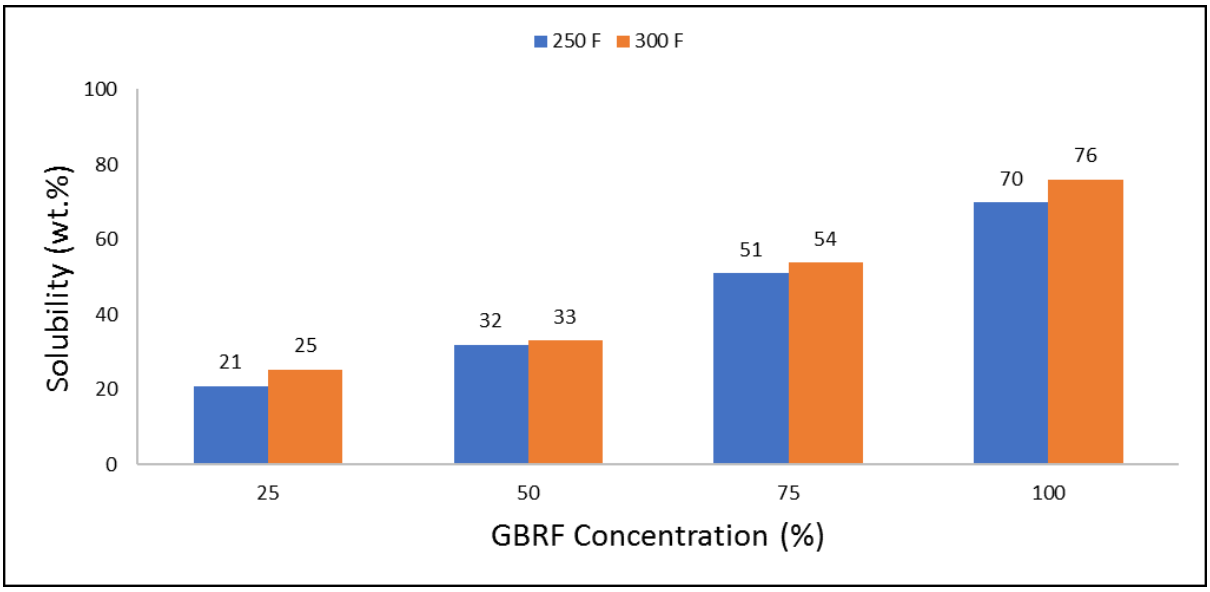

Figure 4-13: The effect of Temperature on Ilmenite Solubility of different wt.% of GBRF at 8 hours.

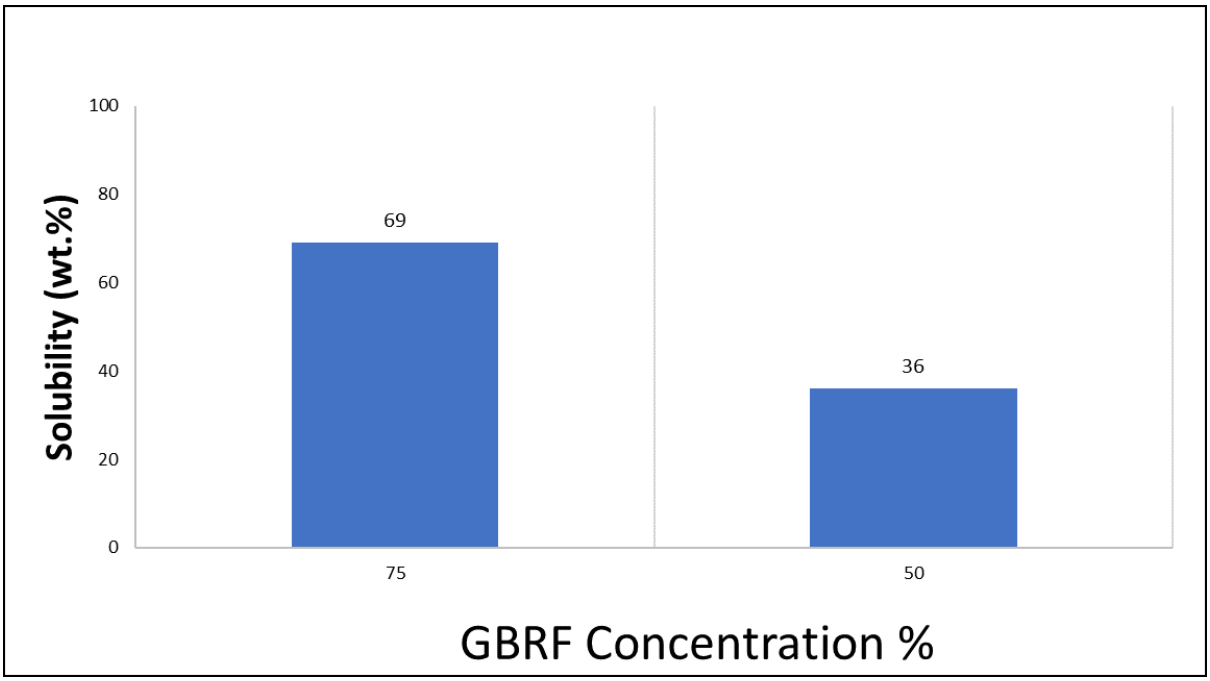

Figure 4-14: The optimized formulation of GBRF 75 wt.% at 24 hours period.

4.2. Fluid Rheology Result

Drilling fluid properties measurements at different temperature were conducted evaluate the fluid properties. The comparison tests showed the expected behavior which is drilling fluid properties decreased when the temperate and pressure increased. The plastic viscosity was decreased, the gel strength was decreased, and yield point were decreased as well. Table 4-1, Table 4-2, and Table 4-3 illustrates the ilmenite drilling fluid rheology at 80°F,200°F,250°F, respectively. Figure 4-15show the comparison of shear stress of ilmenite mud with different temperatures.

Table 4-1:Ilmenite Drilling Fluid Properties 80 °F

Property	Unit	Range
Density	Pcf	98
Plastic Viscosity	cP	29-35
Yield Point	Lb/100 ft^2	45-55
Gel Strength, 10 sec		12
Gel Strength, 10 min		15
pH	-	11

Table 4-2: Ilmenite Drilling Fluid Properties 200 °F

Property	Unit	Range
Plastic Viscosity	cP	22
Yield Point	Lb/100 ft^2	36
Gel Strength, 10 sec		5
Gel Strength, 10 min		8

Table 4-3: Ilmenite Drilling Fluid Properties 250 °F

Property	Unit	Range
Plastic Viscosity	cP	18
Yield Point	Lb/100 ft^2	26
Gel Strength, 10 sec		5
Gel Strength, 10 min		7

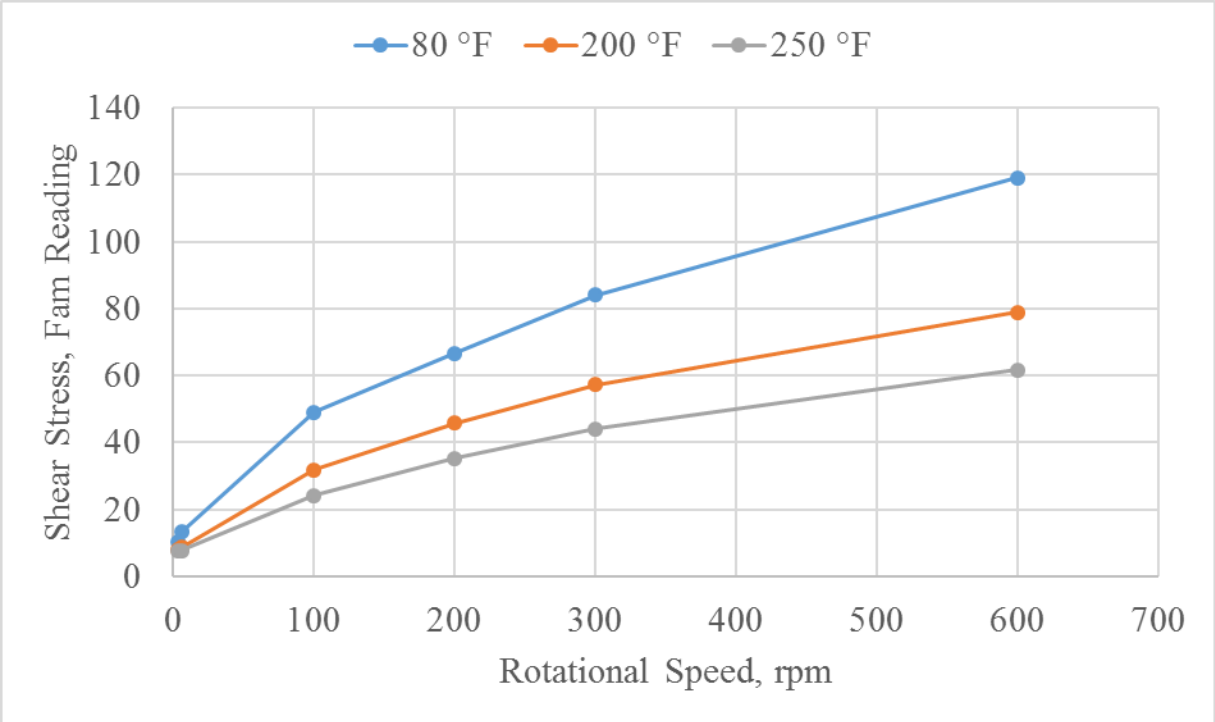

Figure 4-15: Comparison of shear stress of ilmenite mud with different temperature.

4.3. HPHT Filtration and Removal Result

Figure 4-16 shows that the cumulative filtrate volume was high (24 cm^3) when using (1:2) ratio of calcium carbonate (fine; 25 µm and medium; 50 µm, respectively). This introduced a filter cake of 0.4 in. thickness as shown in Figure 4-17. Therefore, it was decided to increase the quantity of medium calcium carbonate to be equal to the fine one to reduce the volume filtration.

Figure 4-18 illustrate the filtration result of three different Indiana limestone. The maximum volume of filtration was 14 cm^3 and the thickness of filter cake was 0.21 in.

from previous static HPHT filter press test. These results show that current formula of

water-based drilling fluid has a good fluid loss control behavior under static conditions.

Table 4-4 and Figure 4-18 show the HPHT summary for the limestone core samples and the filter cake formed by the ilmenite drilling fluid before removal.

In removal part, the formed filter cake was soaked with 300 g solution, which contains 75 wt.% GBRF, 6wt.% corrosion inhibitor, and 6wt.% intensifier. The removal test was conducted at the same HPHT condition and it was run for 24 hours. Figure 4-20shows the filter cake remaining on the core surface after removal test.

The removal efficiency was very excellent using the GBRF solution. It reaches up to 96% of the formed filter cake. The retained permeability was calculated as well for the three samples and did show very good result. The GBRF solution was not only able to removal filter cake but also be able to enhance the core sample. Table 4-5 and summaries the removal efficiency and retained permeability result. However, there is a difference in ilmenite removal efficiency between solubility test and HPHT filtration test and this discrepancy because of the presence of other additives in completion fluid.

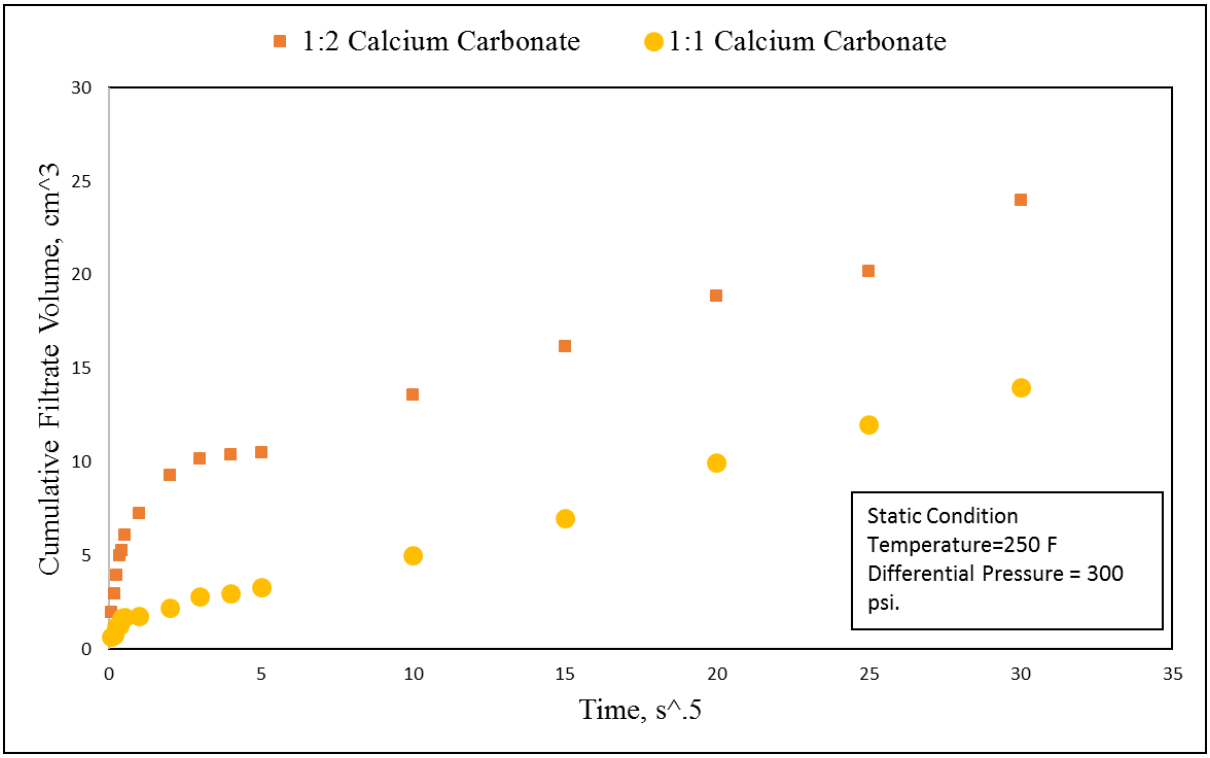

Figure 4-16: The effect of increasing Calcium Carbonate (medium size) the filtrate volume.

Figure 4-17: 0.4 in filter cake formed by using (1:2) ratio of medium and fine CaCo3

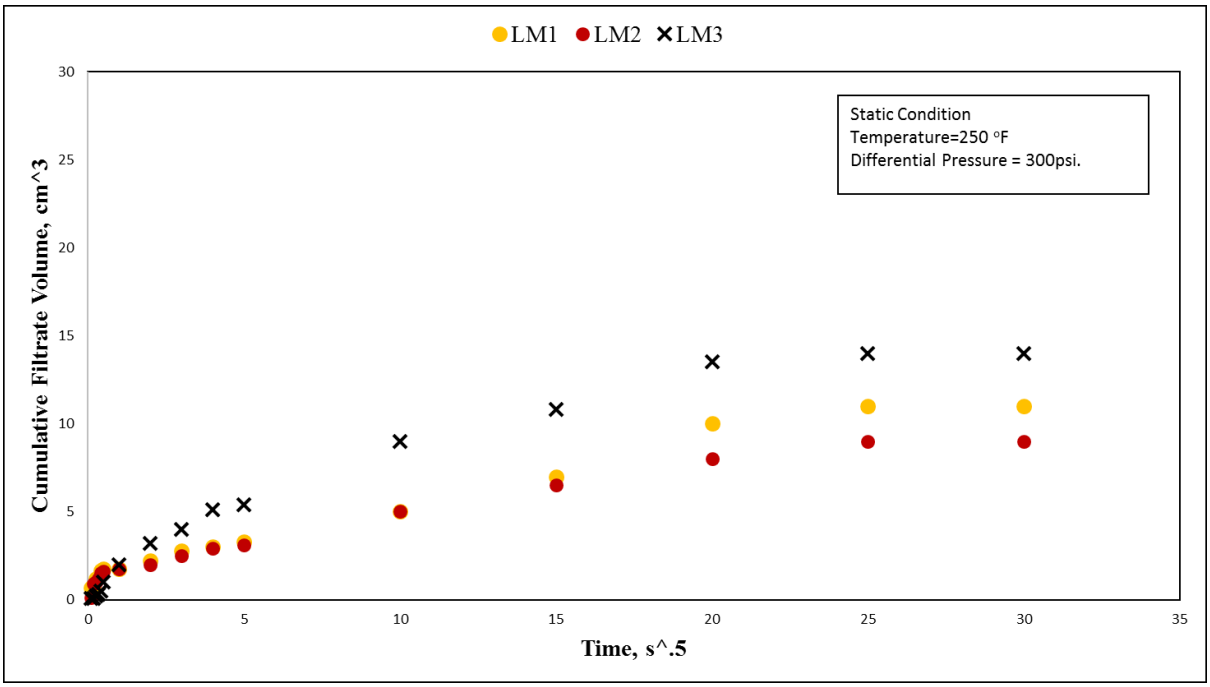

Figure 4-18: Filtration Performance of Ilmenite for three different Indiana limestone samples

Table 4-4: HPHT Filtration Test Summary

Code	Porosity, %	Filtrate, cm3/30min	Filter-cake Weight, g	Filter-cake thickness, in
LM1	11%	9	63.8	0.20
LM2	14%	8	37.7	0.19
LM3	12%	14	41	0.21

Figure 4-19:Filter-cake Before Removal

Figure 4-20: Filter-cake After Removal

Table 4-5: Removal Test and Retained Permeability Summary

Code	Initial Permeability, mD	Final Permeability, mD	Removal Efficiency, %	Retained Permeability, %
LM1	30	33	96.08	110
LM2	15	15	96.82	100
LM3	14	13.9	91.46	99.1

CHAPTER 5
CONCLUSIONS

5.1. Summary

In this study, a series of experiments, including ilmenite solubility test, the drilling fluid rheological properties, HPHT filtration test, HPHT removal test, and Retained permeability measurements were conducted to examine the effect of new formula system solution to remove ilmenite filter cake. The following conclusions can be made because of the previous experiments:

- Chelating agents such as GLDA, ETDA, and DTPA did not dissolve ilmenite very well.
- Catalysts such as Potassium carbonate, potassium hydrogen carbonate, and potassium chloride could not dissolve ilmenite at 300 ^{o}F.
- GBRF solution show up to 96% of ilmenite dissolution rate for solubility test at a period of 24 hours.
- Filtrate volume generated by filtration test on ilmenite water-based drilling fluid was in accepted range (< 14 cm^3) for the HPHT (250 ^{o}F, 300 psi).
- The GBRF solution (75wt.% GBRF, 6wt.% corrosion inhibitor, 6wt.% intensifier) was able to dissolve 96% of the filter cake created by water-based drilling mud weighted with ilmenite particles.
- The new removal system did show excellent retained permeability of Indiana limestone core samples with an average of 100%.

5.2. Recommendation

More experiments could be done to evaluate the new removal technology for other mud types like oil-based mud and all-oil mud, and the results could be compared with water-based mud. In addition to that, different type of rock such as sandstones could be test using the same removal fluid. A fluid to solid ratio can be studied in order to avoid any excessive in fluid removal volume.

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